Development of Flame Retardant Green Composites by Hybrid Flame Retardant Approach

23rd International Conference on Composite Materials

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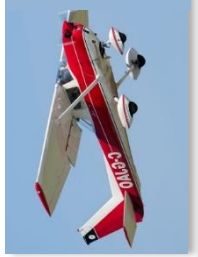
Natural Fiber Composites - Scenario

Initial stage

Less importance, demand & awareness

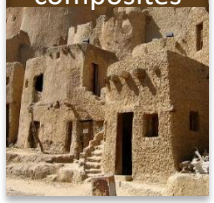
Flax + Phenolic: Fuselage skins of spitfires — 1939



Hemp: Prototype car by Henry Ford — 1942

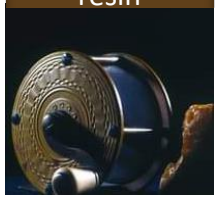
Cotton + PE: Body of "Trabant" car in Germany — 1950-90

Clay/Straw reinforced composites — 1500 BC



Composite bow — 1200 AD

Parkesine – 1st synthetic resin — 1870's



Propagation stage

Slowly started for commercial applications

Flax, Hemp: Number of automotive applications — 2000~

Kenaf + PLA: Cases of cellular phones — 2004~

Flax, Hemp Kenaf: Sporting Good — 2006~

Hemp, Kenaf: Thermal and acoustic insulation panel — 2010~

Present scenario

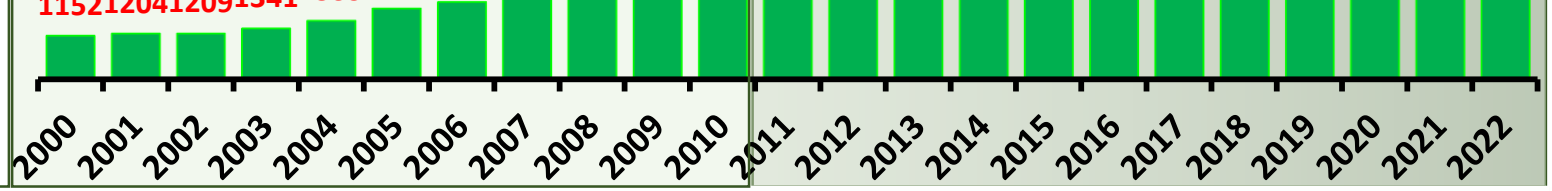
Slowly emerging for commercial applications

Motorsports

Furniture & Interior

Automotive interior

Skin & Sporting



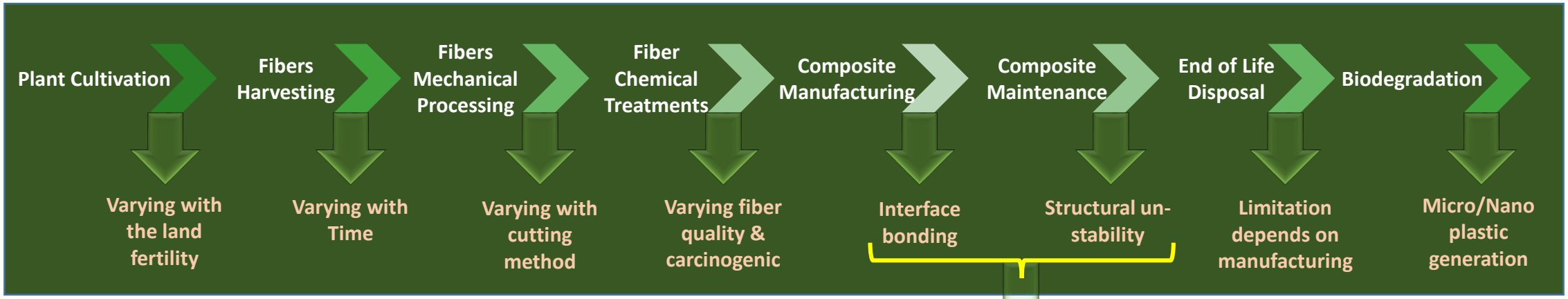
Number of article published on Natural Fiber Composites (NFC), Green Composites and Fire Retardancy of NFC in Elsevier journals. www.sciencedirect.com (December 2022)

1. INTRODUCTION

NFCs - Limitations

While natural fibers offer a sustainable and renewable alternative to carbon or glass fiber, the composites industry has been slow to adopt them due to various challenges.

Limitations: Main stages of Natural fibers – Natural Fiber Composites



J. Marine Science and Engineering 2023, 11, 1076

Limitations - Natural Fiber Composites – Commercialization

- ✓ Limited supply and Variable fiber quality
- ✓ Limited mechanical strength in finished parts
- ✓ Differences in manufacturability and depending on the material
- ✓ Higher material cost compared with fiber glass

Keen Issues – Current Research Focus

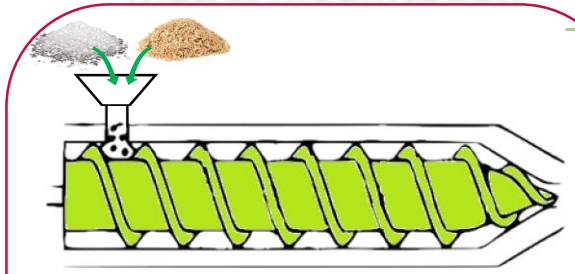
- ✓ Thermally weak and sensitive to flame
- ✓ Mechanical stability in the presence of FR additives

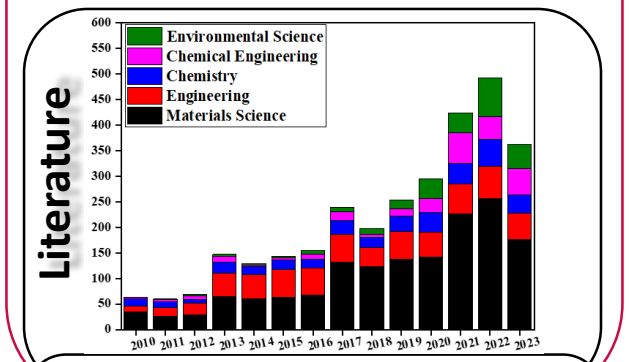
1. INTRODUCTION

NFCs – Developments & Objectives

Fabrication of flame resistant Bio-composites

Matrix modification





Drawbacks

- ✓ Influence composite strength
- ✓ Higher loading for effective
- ✓ Increase viscosity
- ✓ Complexity In Mixing

FOCUS

Hybrid approach

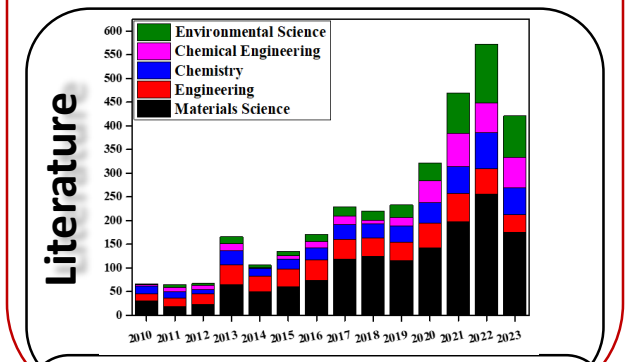
Modifying both Matrix and Reinforcement

Eco-friendly materials

fire resistance

Manufacturing of **Flax fiber** reinforced **Vinyl ester** composite

Reinforcement modification



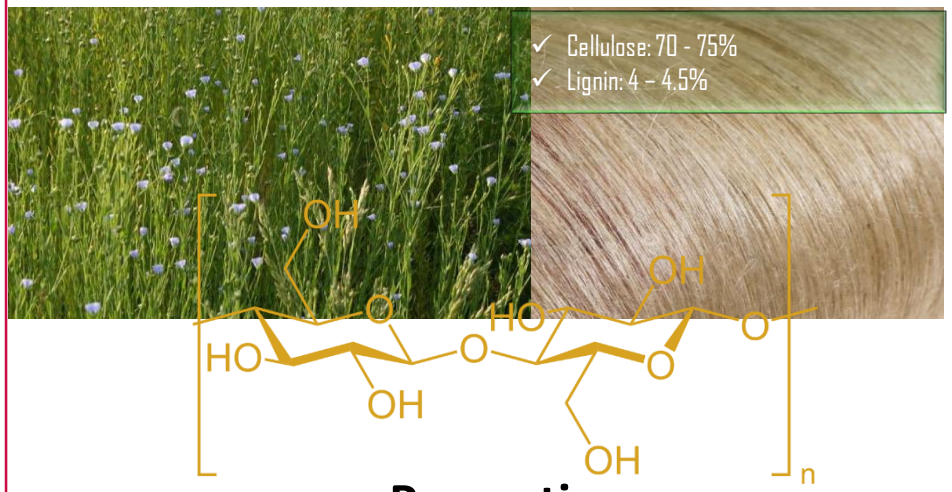
Drawbacks

- ✓ Involve in curing/processing chemistry
- ✓ Lot of waste during treatment
- ✓ Making FR solution is complex

2. EXPERIMENTAL & METHODOLOGY

Reinforcement: Flax fibers

Cellulose Structure



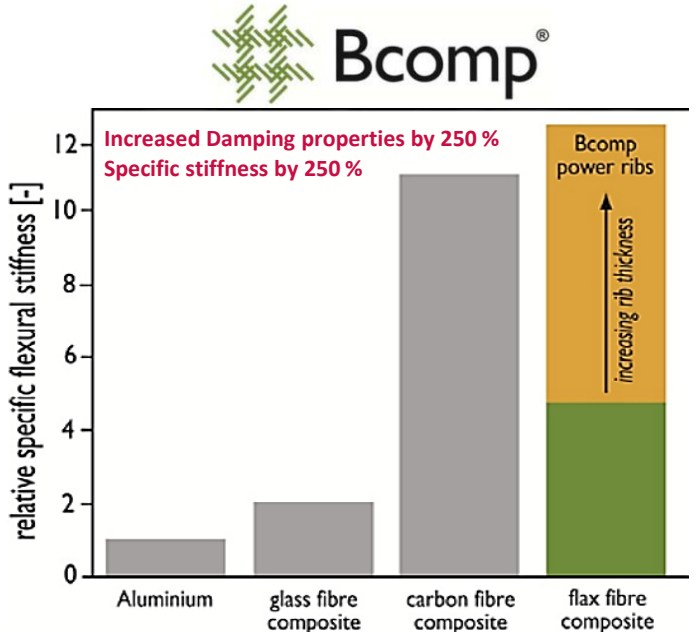
Properties

Fibers	E-glass	Flax	Hemp	Jute	Kenaf
Density (kg/m³)	2550	1530	1520	1520	1193
E-modulus (GPa)	71	58±15	60	60	14-38
Tensile strength (MPa)	3400	1339	920	860	240
Elongation at break (%)	3.4	3.27	1.7	2	-

Materials (Basel) 6(11), 5171, 2013

Advantages

- ✓ Better Mechanical Properties
- ✓ Water absorption in small (7%)
- ✓ Flax fiber has a structure which can be compared to a composite material composed of helical cellulose fibrils embedded in a matrix of polymer (hemi-cellulose, lignin and pectin)



www.BCOMP.CH

Dis-Advantages

- ✓ Flammable
- ✓ Dimensional instability
- ✓ High moisture absorption
- ✓ Anisotropic behavior
- ✓ Limited processing temp.
- ✓ Fugal attach and microbial

Low Limited Oxygen Index

	LOI	HRR
PP/Kenaf	19	700 kW/m²
PP/Flax	21.6	731 kW/m²
Epoxy/Flax	21.3	709 kW/m²

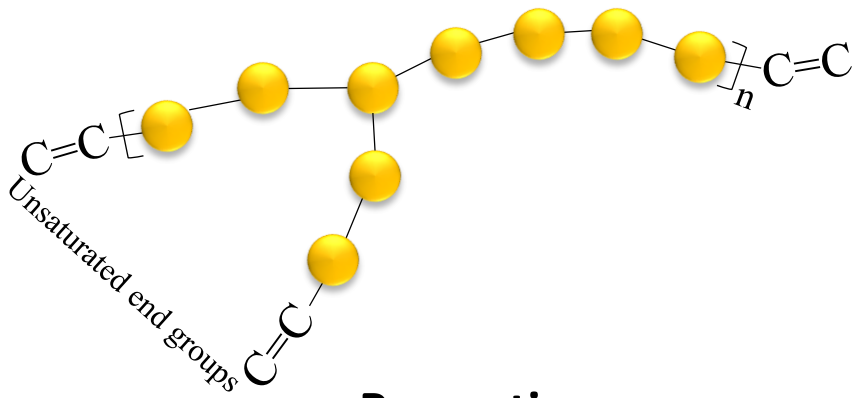
Composites Science Technology 162,2018

Flax Fabric

2. EXPERIMENTAL & METHODOLOGY

Matrix: Vinyl ester

Structure



Properties

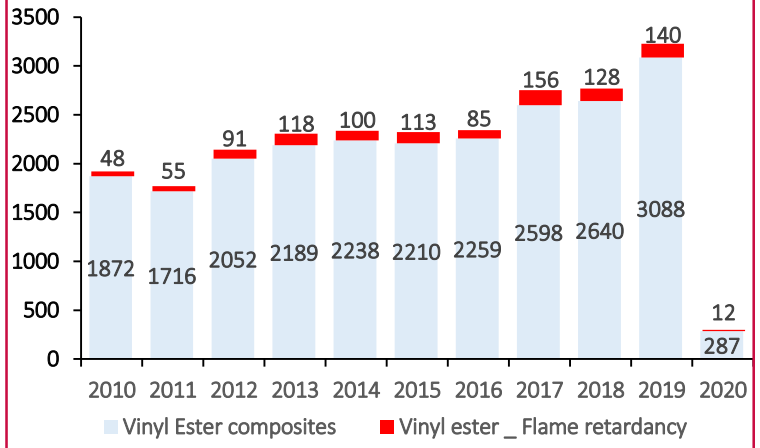
Property	Polyester	Vinyl ester	Epoxy
Density (g/cm^3)	1.2-1.5	1.2-1.4	1.1-1.4
Tensile strength (MPa)	40-90	69-83	35-100
Elastic Modulus (GPa)	2-4.5	3.1-3.8	3-6
Elongation (%)	2	4-7	1-6
Izode impact strength (J/m)	0.15-3.2	2.5	0.3
Compressive strength (MPa)	90-250	100	100-200
Water absorption (24h @20 °c)	0.1-0.3	0.1	0.1-0.4
Cure shrinkage (%)	4-8	N/A	1-2

Composites: Part B 56, 296, 2014

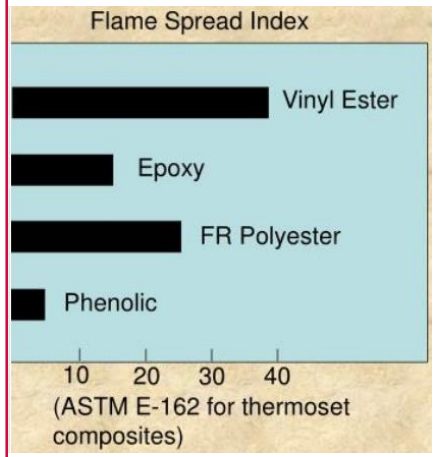
Advantages

- ✓ Most of the properties of Vinyl ester are intermediate between Polyesters and Epoxies
- ✓ Some of the most important properties are:
  - Water and chemical resistance
  - Electrical Stability
  - Thermal stability
  - Toughness
  - Low volatiles during manufacture
  - Low shrinkage
  - Average cost

Literature

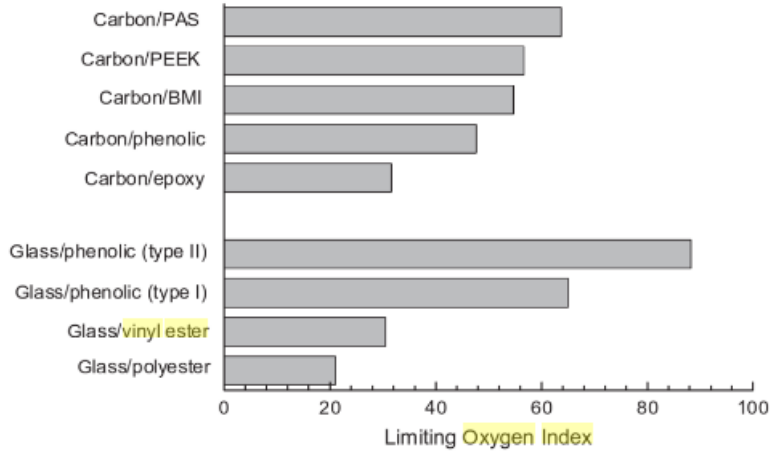


Dis-Advantages



- ✓ High shrinkage
- ✓ Poor fire resistance
- ✓ Higher cost than polyester

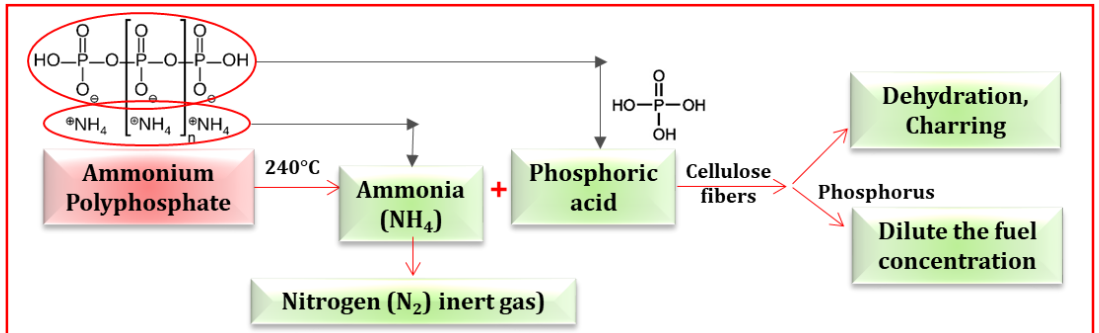
Vinyl ester panel



2. EXPERIMENTAL & METHODOLOGY

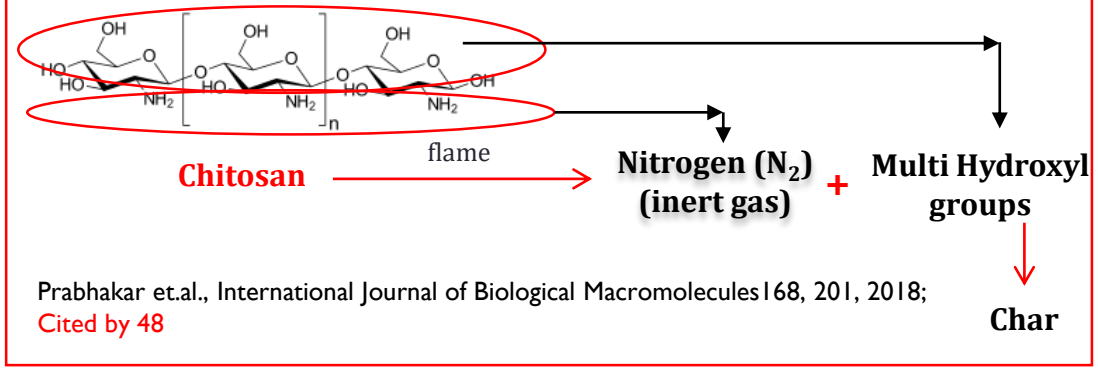
Flame Retardants: CS, APP & NCSAPP

Chitosan (CS)



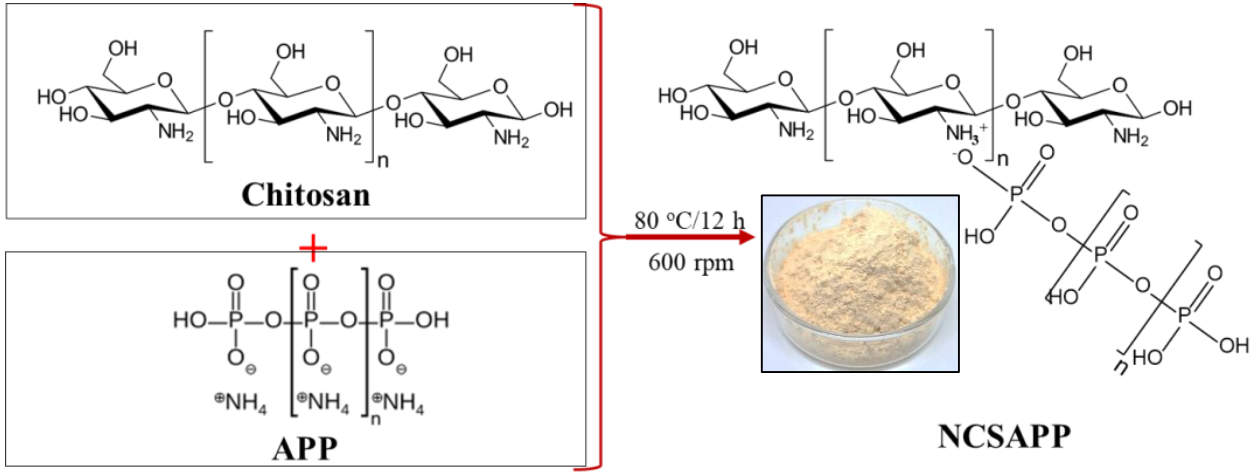
Prabhakar et.al., Carbohydrate Polymers 168, 201, 2017; Cited by 64

Ammonium Polyphosphate (APP)



Prabhakar et.al., International Journal of Biological Macromolecules168, 201, 2018; Cited by 48

Novel Flame Retardant (NCSAPP)



Prabhakar et.al., Cellulose 26, 8801, 2019; Cited by 47

The synthesized NCSAPP was found to exhibit superior fire retardant properties than CS and APP.

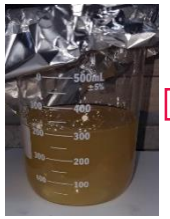
2. EXPERIMENTAL & METHODOLOGY

Manufacturing of Vinyl ester/Flax fabric

Flame retardant filler (5 wt.%)

Chitosan APP NCSAPP

Vinyl ester



Mixed solution

Resin+Curing agents

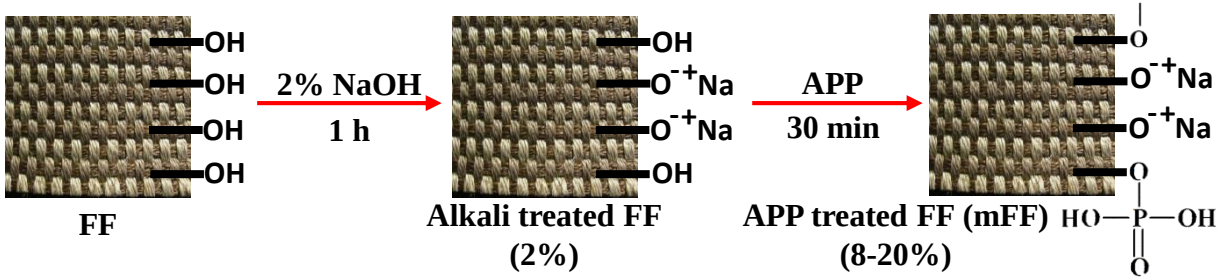
Magnetic Stirring

+

Three roll mill

Mixing method

Surface modification of Flax Fibers

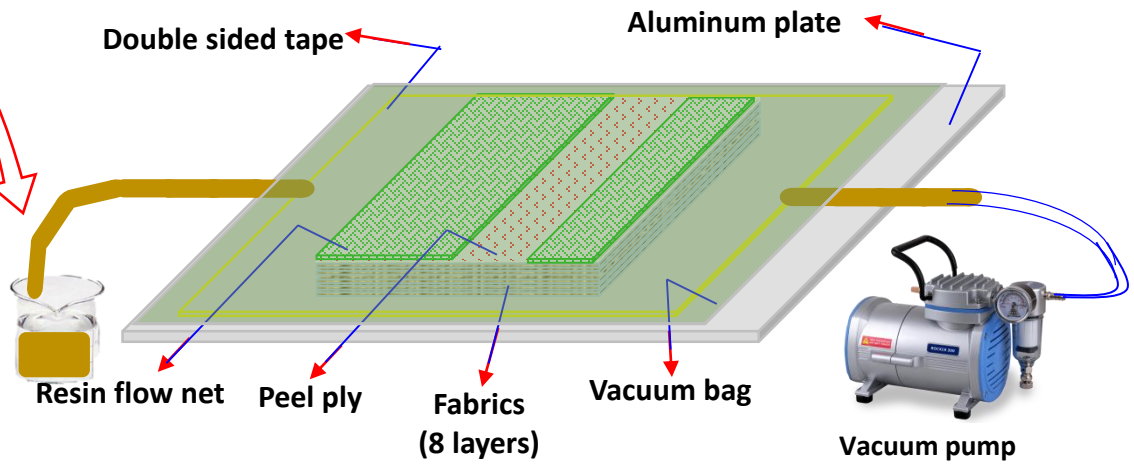


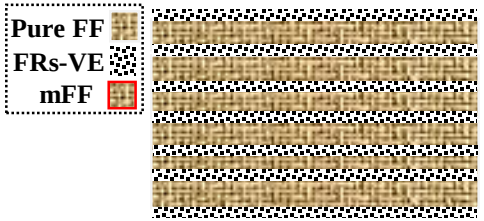
FF

Alkali treated FF (2%)

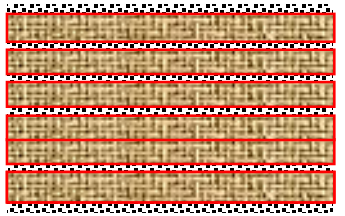
APP treated FF (mFF) (8-20%)

VARTM process





FRs-VE/mFF Composite



FRs-VE/FF Composite

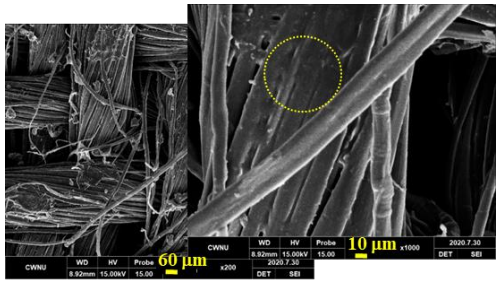
Modified Flax Fiber

3. RESULTS & DISCUSSION

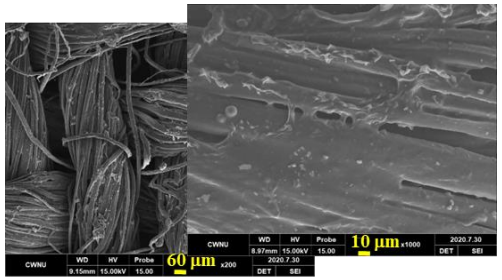
APP treated FF – Surface Morphology & X-RD

Surface Morphology

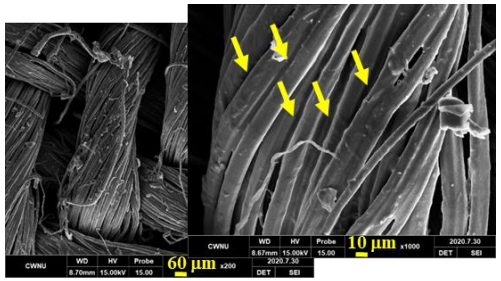
Flax fibres



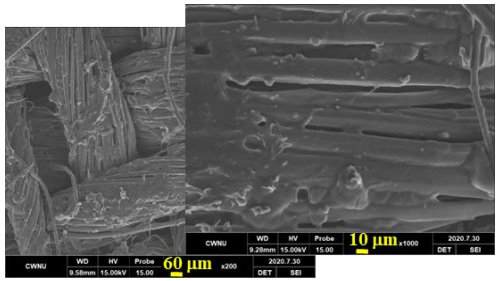
10%APP treated Flax fibres



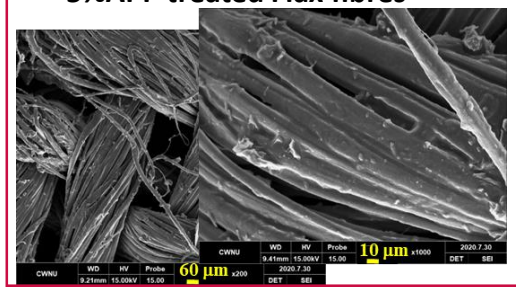
Alkali treated Flax fibres



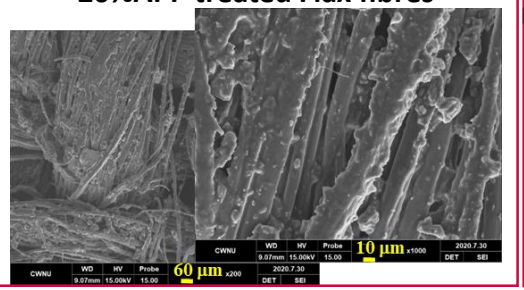
15%APP treated Flax fibres



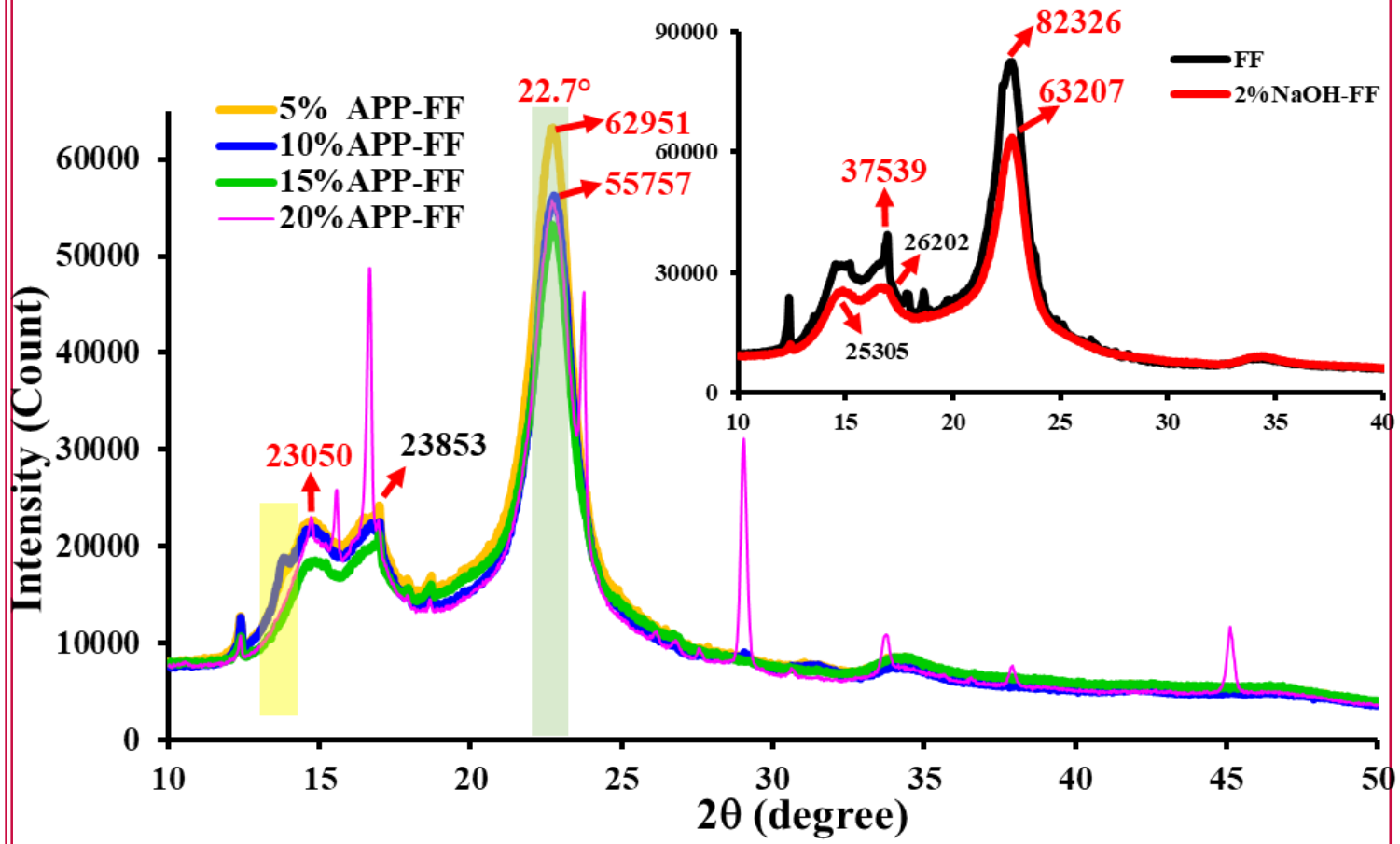
5%APP treated Flax fibres



20%APP treated Flax fibres



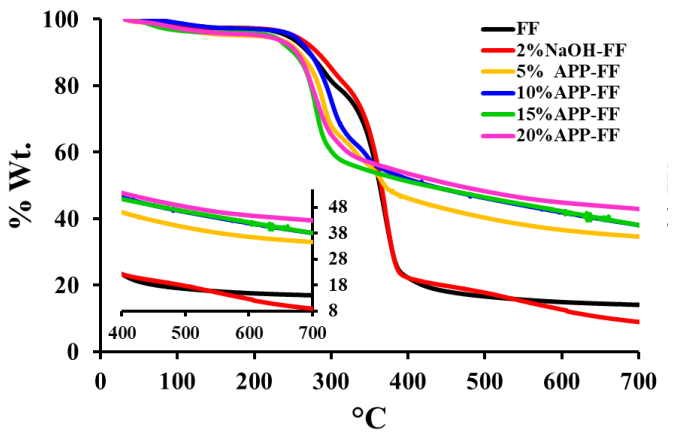
X-ray patterns



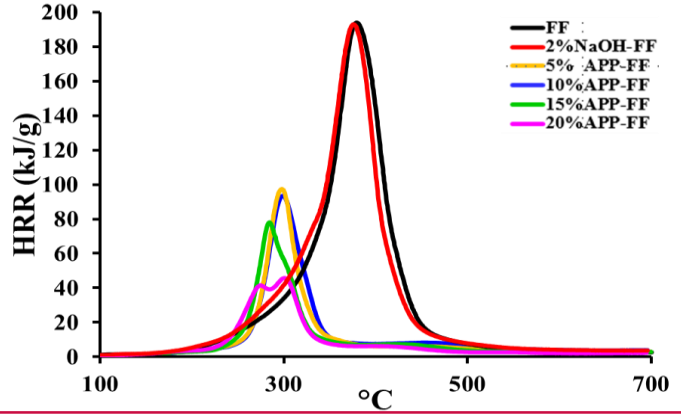
3. RESULTS & DISCUSSION

APP treated FF – Thermal & Flame retardancy

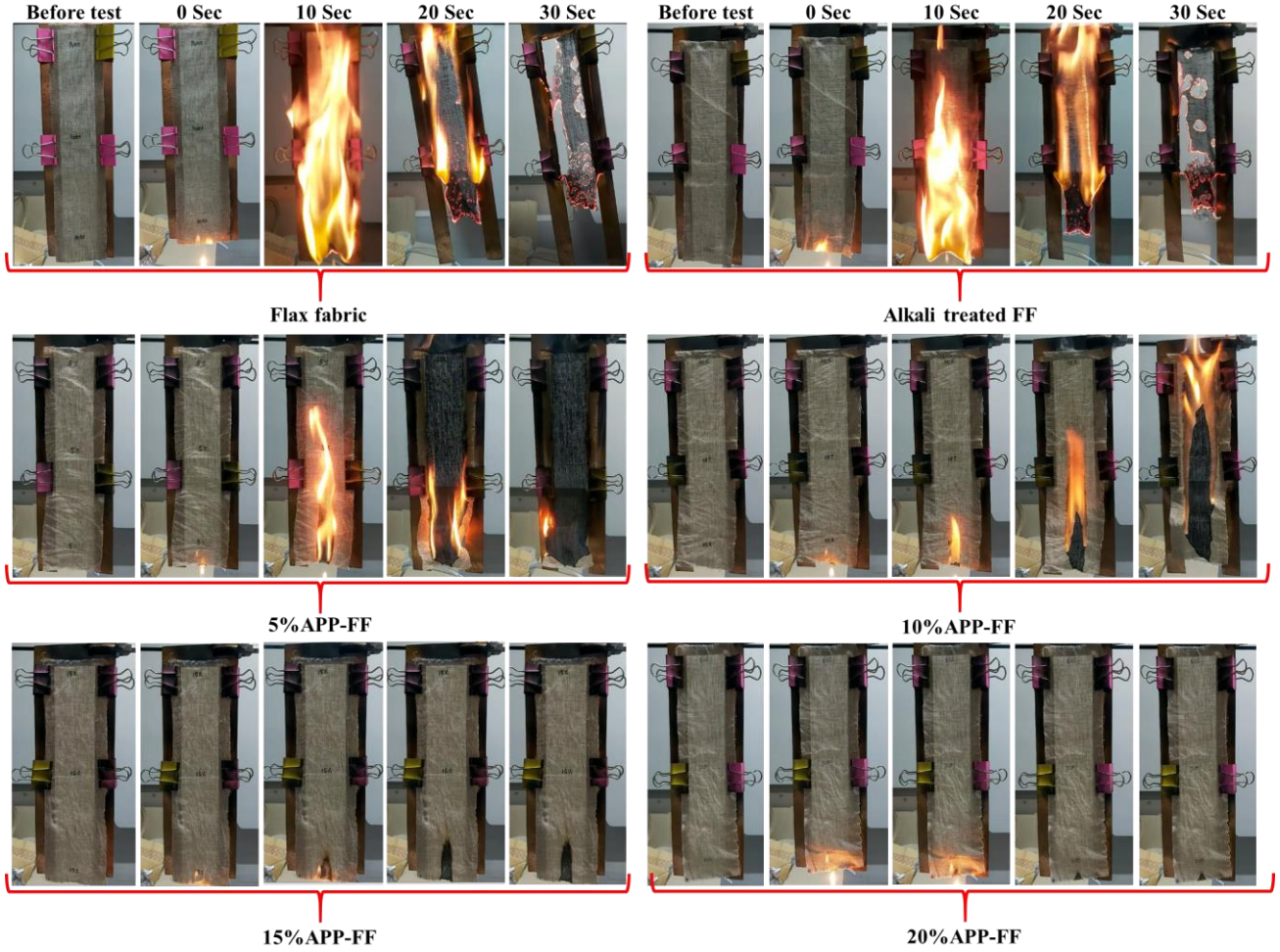
Thermogravimetric analysis



Pyrolysis combustion



UL-94 Vertical flame test

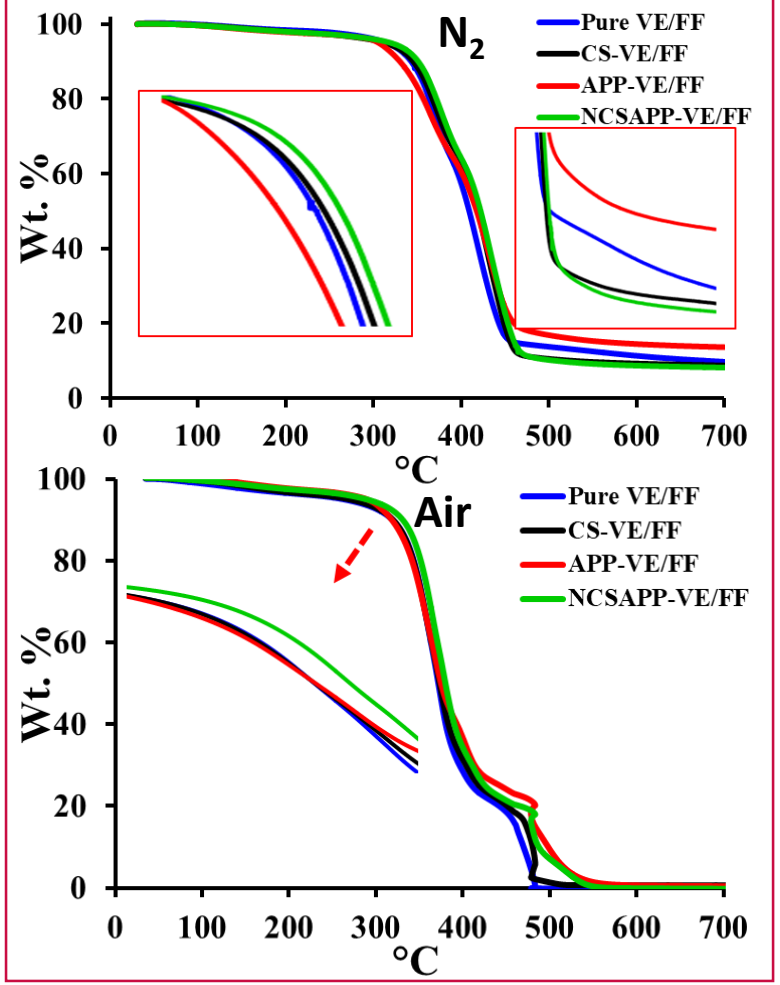


FRs loaded Vinyl Ester/Flax Fiber Composites

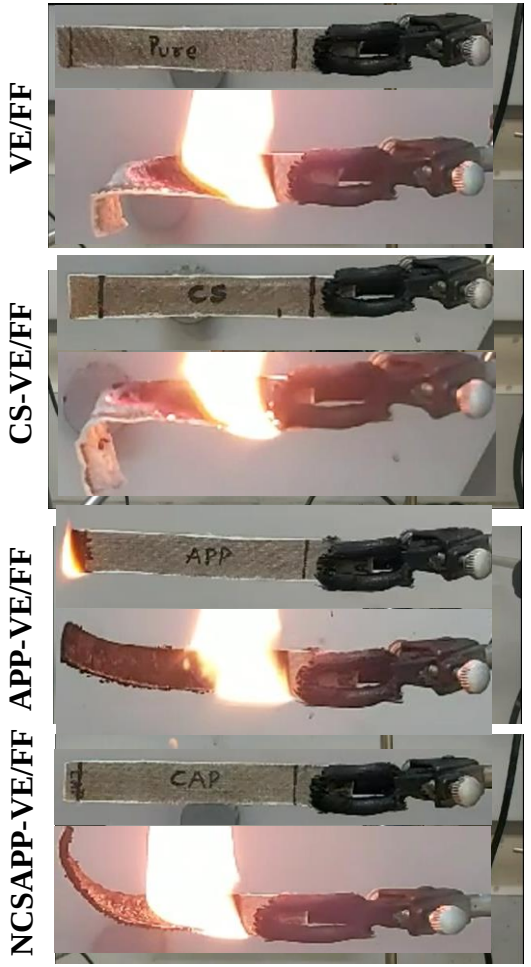
3. RESULTS & DISCUSSION

FRs-VE/FF Composite – Thermal & Flame retardancy

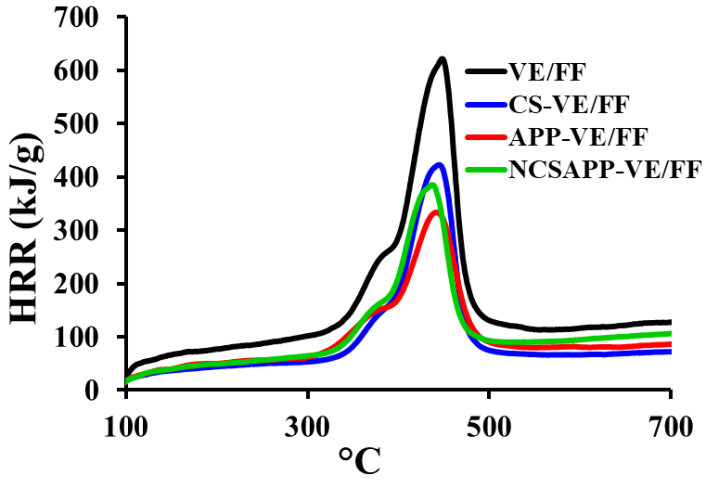
Thermogravimetric analysis



UL-94 Vertical flame test



Pyrolysis combustion



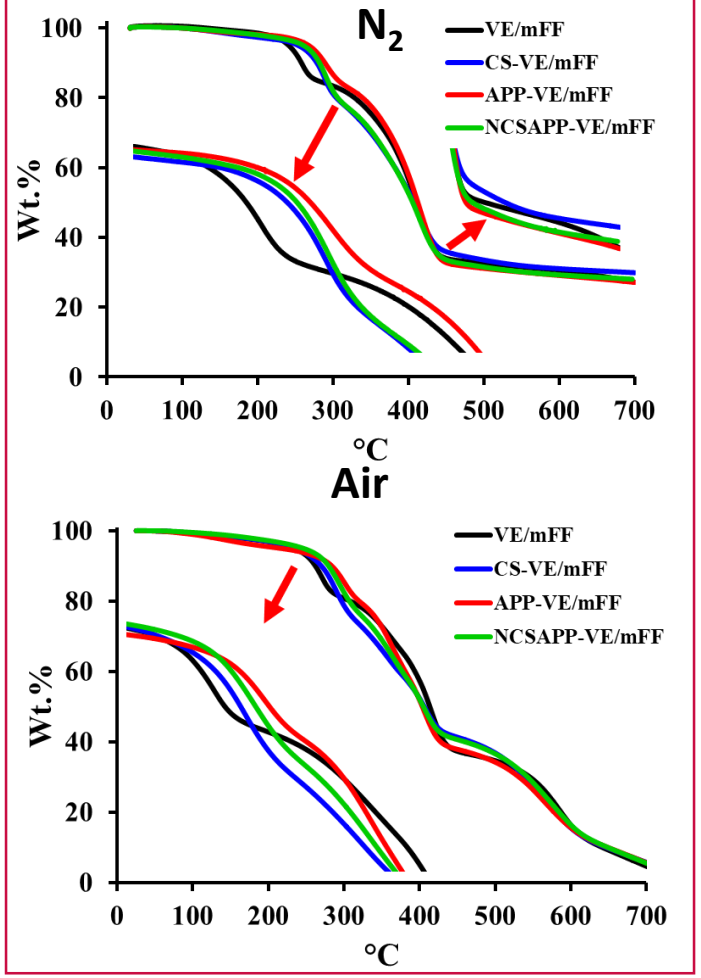
Sample	Horizontal Flame Test		Micro calorimeter	
	Air		O_2+N_2	
	Burn time [min]	Burn rate [min/sec]	T [°C]	pHRR [w/g]
VE/FF	4:05	0.30	380.31	446.0
CS-VE/FF	3.95	0.31	422.66	445.3
APP-VE/FF	3.30	0.37	333.32	443.4
NCSAPP-VE/FF	4.28	0.29	385.25	449.2

FRs loaded Vinyl Ester/Modified Flax Fiber Composites

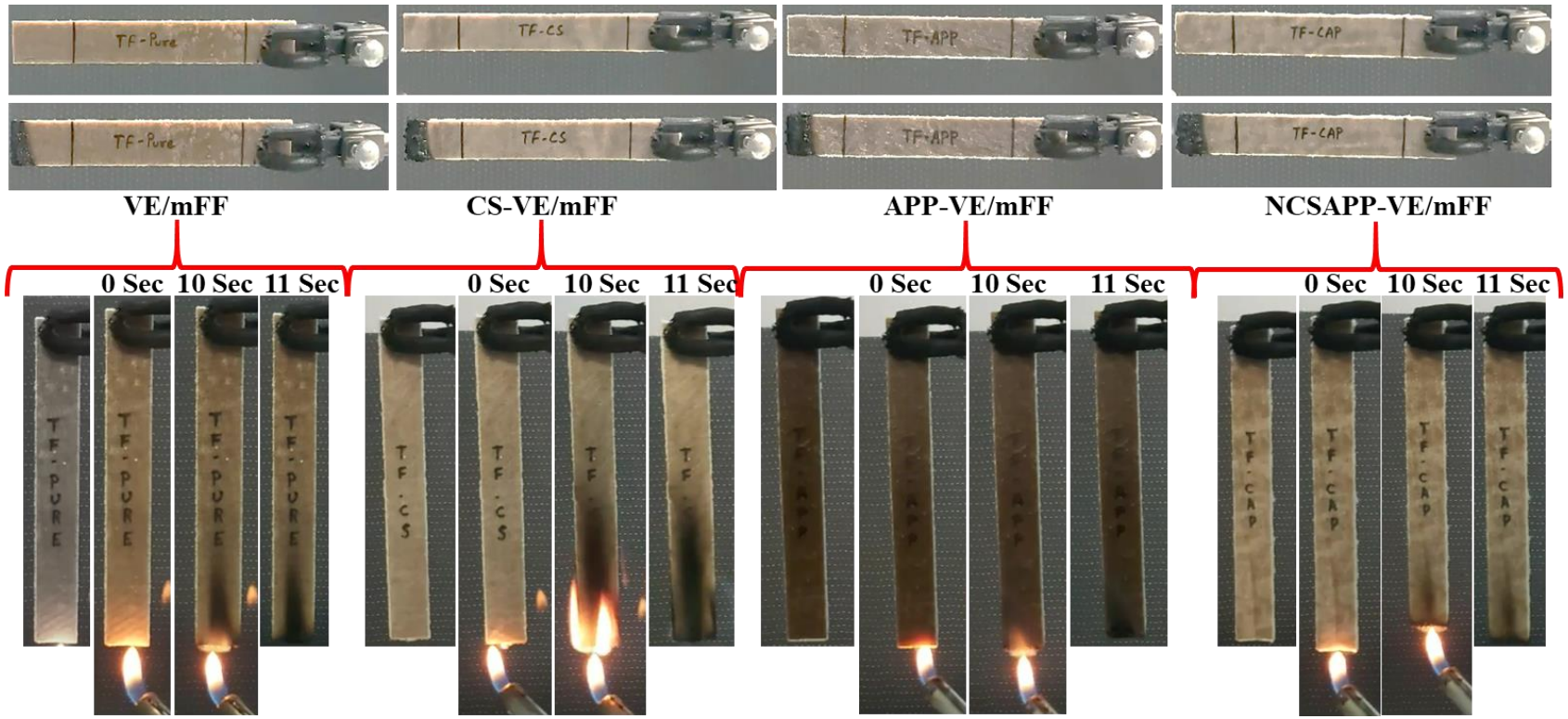
3. RESULTS & DISCUSSION

FRs-VE/mFF Composites– Thermal & Flame resistance

Thermogravimetric analysis



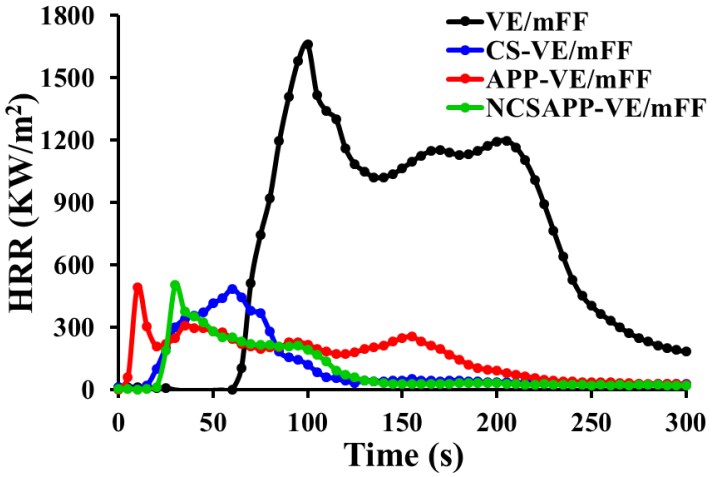
UL-94 Horizontal & Vertical flame test



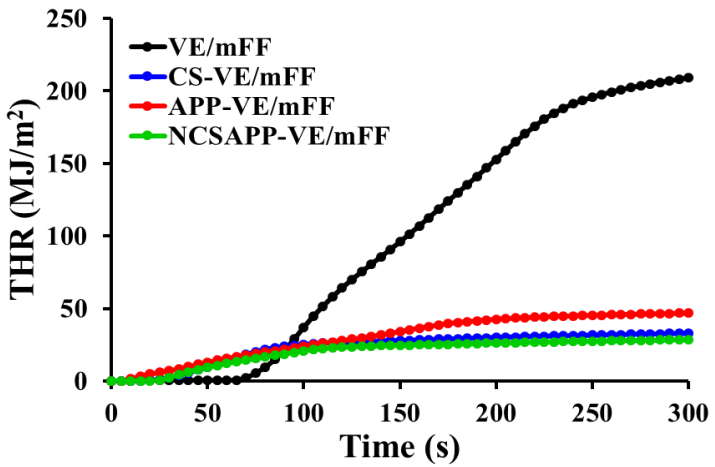
3.RESULTS & DISCUSSION

FRs-VE/mFF Composites– Flame resistance

Heat Release Rate (HRR)

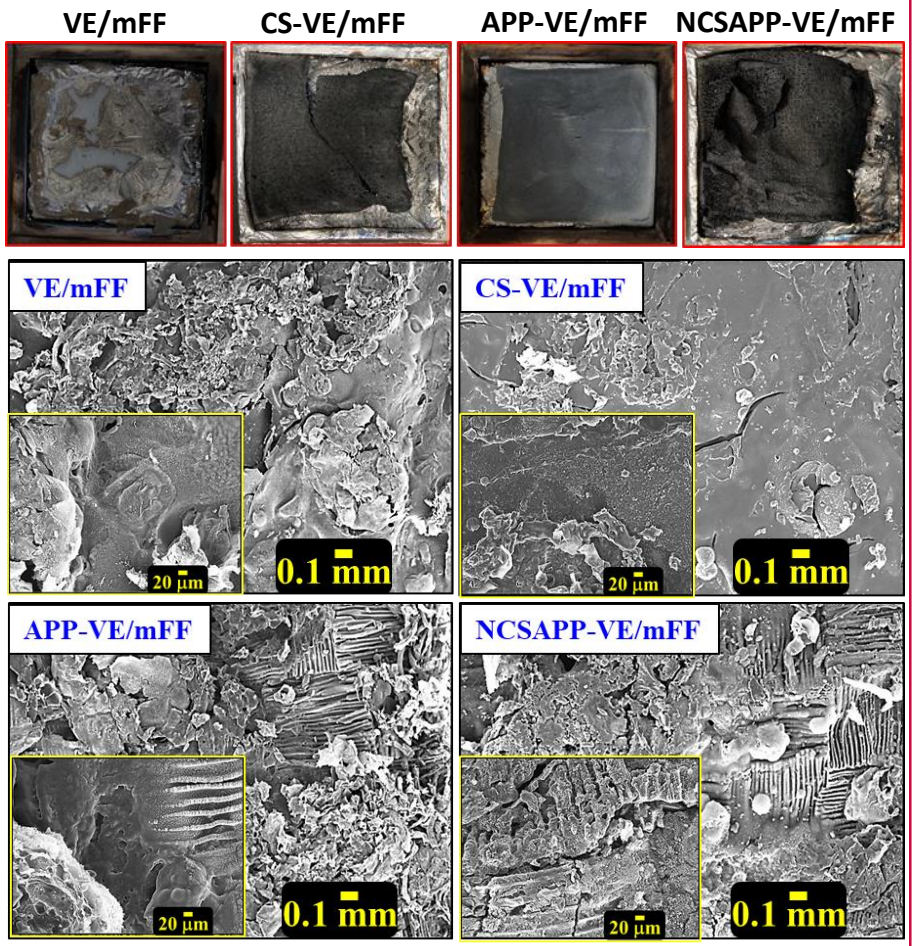


Total Heat Release Rate (THR)



Sample	VFT	Cone calorimeter					
	Air	Air					
	Rank	pHRR [KW/m²]	THR [MJ/m²]	TSR [m²/m]	CO_2 P [g/s]	CO P [g/s]	SPR [m²/s]
VE/mFF	V0	1659	248.7	10882	0.84	0.068	0.67
CS-VE/mFF	V0	483	33.1	1212	0.29	0.027	0.20
APP-VE/mFF	V0	493	53.8	2633	0.17	0.017	0.22
NCSAPP-VE/mFF	V0	503	28.6	1421	0.26	0.025	0.20

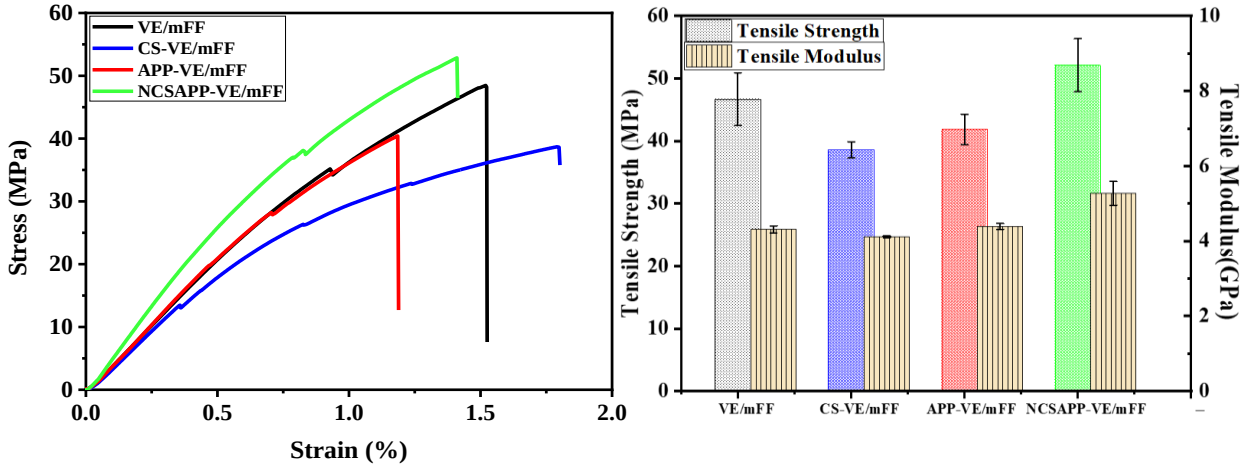
Char morphology



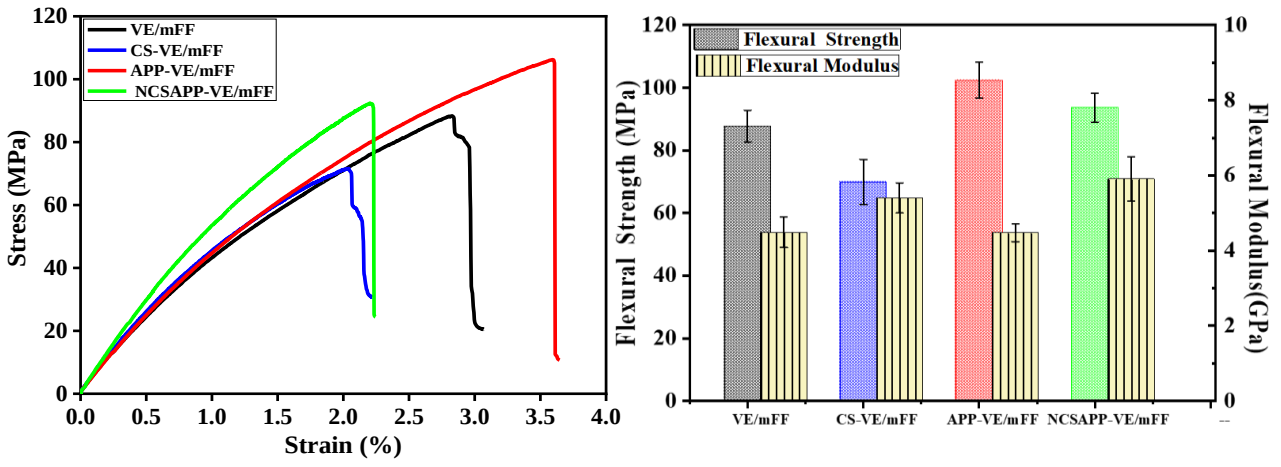
3. RESULTS & DISCUSSION

FRs-VE/mFF Composite – Tensile & Flexural Behavior

Tensile behavior



Flexural behavior

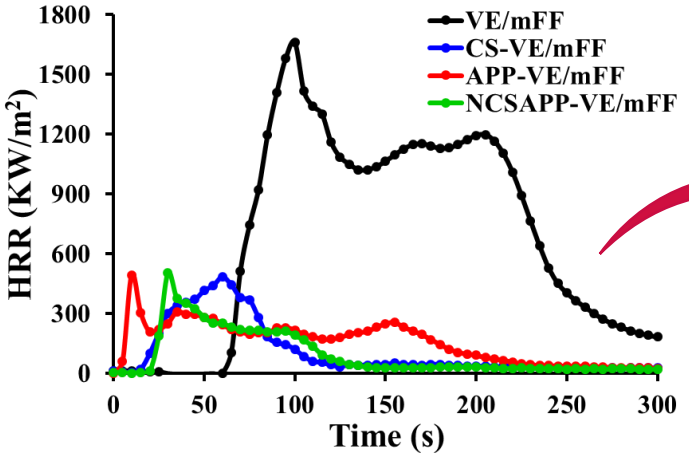


- ✓ **The porosity of the fibers was reduced owing to the settlement of the APP between the gaps of the woven folds.**
- ✓ **The change in the fiber surface reduced the interaction between the resin and the fibers, thereby reducing the strength of the pure and FR-filled composites.**
- ✓ **Generally, the compatibility of the additives with a matrix/reinforcement determines the mechanical properties of the composite. An additional advantage of this approach is that a single compound can be used for the additive and surface treatment.**

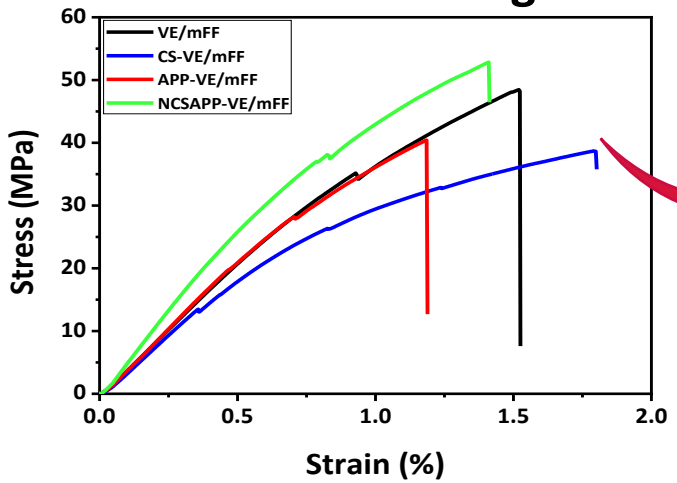
4. CONCLUSIONS

VE/FF Composites – Application area

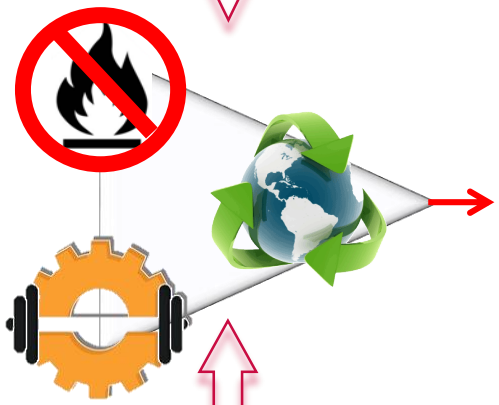
Flame resistance



Mechanical strength



HRR 69%
THR 78%
SPR 75%
CO_2P 65%
COP 60%



TS 10%
TM 18%
FS 14%
FM 24%

Probable application area

Outdoor high temperature technical products

Automotive headliner, Parcel shelf, Engine under cover

Interior and Exterior

Sub-structural components

4. CONCLUSIONS

VE/FF Composites

- Flame resistant Vinyl ester/Flax fiber composites are manufactured hybrid approach.
  - ✓ The APP-modified FF exhibited excellent thermal stability (38% residue at 700 °C) and flame retardancy (UL94-V0 and HRR-77.64 w/g).
  - ✓ VE/modified FF and FRs filled VE/modified FF composites exhibited significantly higher thermal stability owing to the formation of ~28% char residue at 700 °C and flame retardancy by reaching V0 rank in UL94, as well as fire extinguishing immediately after removal of the fire source.
  - ✓ The NCSAPP filled VE/modified FF composite showed reasonably better mechanical properties.
- Over all, this investigation provides design for sub-structural components for outdoor engineering applications.

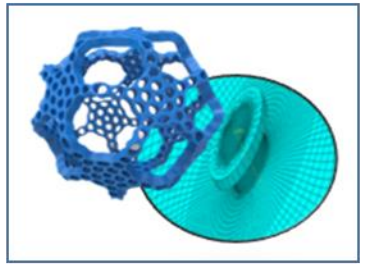
Research Center

Center for Advanced Material Research

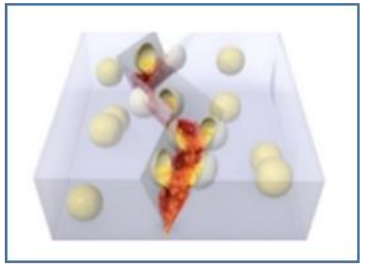
Core Competences

Green Bio-composites

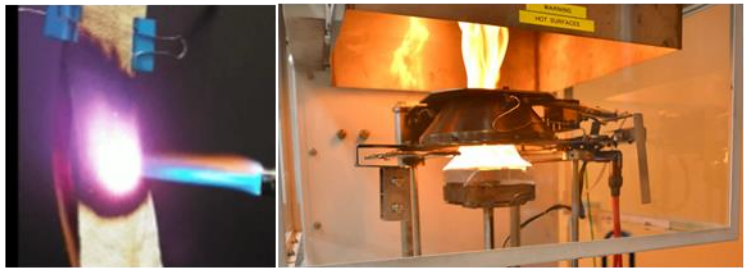
Bio-waste additives



3D printing & Analysis



Self-healing systems



Flame retardant composites

Support Organization	Research Title	Period	Research fund (USD)
NRF National Research Foundation of Korea	Flame resistant Eco-hybrid Composites	2018/06-2027/02	5 million

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Zolbayar

Phuong

Research Center – Natural Fiber Products


Foot guard and Arm guard (Baseball)

Bicycle seat

Tray

Skate board

Fire resist bag

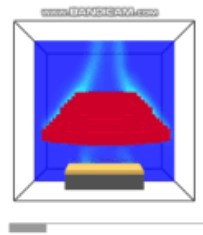
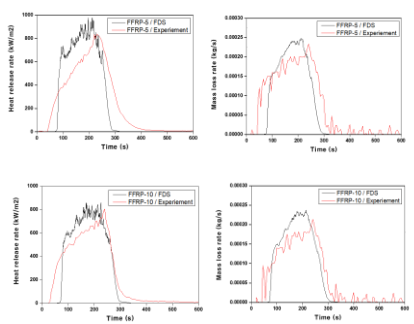
Wind turbine blade

Enough strength for small scale wind turbine

Lightweight

Sound absorption


Flame retardant natural fiber

Predict the combustion behavior of NFRC

Combustion analysis

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Advanced Composites & Structural Design Lab.

CAMR
Center for Advanced Materials Research

ICCM 23 INTERNATIONAL CONFERENCE ON COMPOSITE MATERIALS BELFAST 2023 30 JULY – 4 AUGUST

THANK YOU

For your attention